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NATIONAL BUREAU OF STANDARDS REPORT

7135

THERMAL CONDUCTIVITY OF SEMICONDUCTIVE SOLIDS;
METHOD FOR STEADY-STATE MEASUREMENTS ON
SMALL DISC REFERENCE SAMPLES

Interim Technical Report
Covering Period
February 23, 1959, to March 31, 1961

by

D. R. Flynn and H. E. Robinson

Report to the
Bureau of Ships
Department of the Navy
Washington, D. C.



U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

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Heat Transfer Section
Building Research Division

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THERMAL CONDUCTIVITY OF SEMI-CONDUCTIVE SOLIDS;
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D. R. Flynn and H. E. Robinson

ABSTRACT

An absolute cut-bar method of measuring the thermal conductivity of solids is described, suitable for small specimens of semiconductive materials. Measurements have been made at temperatures from 100° to 800°C on Pyrex, Pyrocera^m, and a nickel-chrome alloy, and representative tentative results are given. The data for the nickel-chrome alloy, over the common temperature range 120° to 600°C, agree with the smoothed data from measurements made on the same material by an independent absolute method, with a standard deviation of 0.7 percent. The data for Pyrex and Pyrocera^m may be subject to errors because of the uncertainties introduced by thermal contact resistance at the surfaces of the specimens.

It is shown that the use of a matched guard in a cut-bar apparatus does not reduce extraneous heat exchanges between the bars and specimen and the surrounding powder insulation to the small magnitude desirable for good accuracy of measurement. A mathematical analysis of the system indicates a superior guarding condition which greatly reduces these extraneous heat flows, and which was used in the measurements mentioned.

Using the knowledge acquired by these investigations, a final model of the apparatus is being built for measurements on small specimens (1/2-in. by 1-in. diameter discs) at temperatures in excess of 1200°C.

1. INTRODUCTION

The thermal conductivity of thermoelectric materials is of great importance in determining the performance of a thermoelectric device. If such a device is to have a high efficiency, the thermal conductivity of the thermoelectric elements must be as low as possible in the temperature range in which they are to operate. Reliable thermal conductivity

data are required at temperatures ranging from room temperature or below to 2000°C, since it is advantageous to operate a thermoelectric generator at as high a temperature as possible.

Reliable thermal conductivity data at elevated temperatures are not generally available, partly because of the difficulties of making such measurements. Many semiconductor materials of interest are available only in small samples because of the difficulty of obtaining them with adequate uniformity. Consequently, it is necessary that a method of measuring the thermal conductivity of such materials be one suitable for small specimens and capable of use at high temperatures.

It is the purpose of this investigation to develop a method and apparatus for steady-state thermal conductivity measurements at temperatures to 800°C and above, and suitable for solids in the form of small specimens (1/2-in. by 1-in. diameter discs), with the objective of providing samples for use by other laboratories as thermal conductivity reference specimens in connection with their measurements on solid semiconductors.

This report discusses the apparatus which is being developed for this purpose and gives a summary of the findings of this investigation to date.

2. METHOD OF APPROACH

2.1 Parameters Involved

The steady-state measurement of thermal conductivity requires a determination of the heat flux through the specimen and of the temperature gradient within the specimen.

For thermal conductivity measurements of the so-called absolute type, the heat flux is determined either by supplying heat to the specimen at a known rate by means of an electrical heater or by calorimetrically measuring the heat flow leaving the specimen. For thermal conductivity measurements of the comparative type, the temperature gradient in a known standard is used to determine the heat leaving or entering the specimen. Since available high temperature insulating materials have a thermal conductivity only one or two orders of magnitude less than that of semiconductor materials, it becomes increasingly difficult to insulate against extraneous heat flows as temperature increases. Thus, the problem is not only to measure the rate of heat input or output, but also to insure that the

heat flow measured coincides with that through the specimen, or that adequate corrections for extraneous heat flows are made.

Because of their small size, thermocouples are generally the most satisfactory elements for measuring temperatures in a specimen or system with minimum disturbance to the heat flow pattern. However, in specimens of small volume, even a thermocouple may introduce some disturbance, and for this reason many investigators have preferred to deduce the temperature at the contact surface of a specimen by extrapolating temperatures observed in the contacting body. If this is done, the difficult problem of the thermal resistance at the contacting interfaces is encountered. In attempting highly accurate measurements, the selection of the best method to use depends upon the parameters of the system, and must be guided by experimental findings.

2.2 Cut-Bar Method

The method most frequently used for determining the thermal conductivity of small solid specimens is a special form of the comparative method, sometimes known as the cut-bar method. The more important features of this design are illustrated in Figure 1. The disc-shaped specimen is interposed between two rods of known thermal conductivity, along which heat is caused to flow by raising the temperature at the far end of one bar. The rod-like assembly is surrounded by an axially concentric guard cylinder; the space between them is filled with a powder-type thermal insulation to restrict heat interchanges. Thermocouples in the "known" bars indicate temperatures from which the heat flow and temperature at each of the ends contacting the specimen can be calculated.

A study of the characteristics of the method led to the following principal conclusions.

(1) A major practical limitation of the general method is that, because of the temperature gradient required in the bar, the maximum mean temperature of the specimen is likely to be one hundred or more degrees C lower than that at the hotter end of the hotter bar. It is undesirable, in view of the importance of high temperature in this program, to have the maximum specimen temperature so much reduced below the maximum apparatus temperature obtainable with the electrical heaters used.

(2) The proper guarding of a cut-bar type of apparatus is discussed in Appendix A. It is shown that the conventional method of guarding such an apparatus (i.e., maintaining the same temperature distribution along the guard cylinder as along the reference bars and specimen, so that guard temperatures match those of the reference bars at corresponding longitudinal positions) does not eliminate extraneous heat exchanges between the reference bars and specimen and the surrounding powder insulation. While it is theoretically possible to place a temperature distribution on the guard which will eliminate parasitic heat flows, to do so would be very difficult. A mathematical analysis, such as that in Appendix A, of a practical compromise guarding condition would serve for evaluating the heat gains or losses into the powder insulation reasonably well. However, these heat exchanges affect the heat flows within the bars, which are used as heat flow meters, and an accurate treatment of their effect requires solution of the four-body composite system involved. Mathematically, this problem is intractable because of the many continuity conditions which must be satisfied. The importance of the problem increases as the conductivity of the powder insulation increases with temperature.

For these major reasons, an alternative method was devised, capable of measurements on disc specimens by an absolute method.

2.3 The Absolute Cut-Bar Method

In order to meet the objections to the cut-bar method given in the preceding section, it was decided to utilize the general cut-bar arrangement, but to make an absolute power measurement rather than to use the bars as references for determining the heat flow. This permits placing the heater much nearer the specimen so that the specimen mean temperature is only slightly lower than the maximum obtainable apparatus temperature. Furthermore, since the bars are no longer used as heat flow meters, the elimination of extraneous heat flows from them is not so critical.

It was decided to build a prototype apparatus first, with which to study the various parameters involved. Figure 2 shows schematically the prototype apparatus as it was first constructed. The disc specimen is interposed between the hot bar and cold bar shown in the figure. The power input to the hot bar heater is accurately measured potentiometrically, and is adjusted to raise the temperature on the hot side of the specimen by the desired amount over that on the cold side. The desired temperature on the

cold side of the specimen is established by regulating the cold bar heater at some temperature in excess of the coolant temperature.

The temperature of the hot and cold guards are controlled by their respective heaters. By varying the guard temperatures, extraneous heat flows can be regulated. (The effect of various guarding conditions is shown in Appendix A.)

Heat flow to or from the hot bar along the hot bar support is prevented by regulating the heater at the base of the support to maintain zero temperature difference between differential thermocouple junctions in the support.

The prototype apparatus was constructed of stainless steel. Calibrated 28-gage chromel-alumel thermocouples were used for temperature measurement. The method of computing thermal conductivity from the data acquired is outlined in Appendix B.

3. RESULTS OBTAINED USING THE ORIGINAL PROTOTYPE APPARATUS

3.1 Description of Pyrex Specimens

Pending availability of zirconia and alumina samples to be furnished, exploratory development tests of the prototype apparatus were conducted using specimens of Pyrex No. 7740. This material was chosen because of its homogeneity and low thermal conductivity, and because some data on its conductivity are available in the literature.

While optically-flat specimens were being prepared, tests were conducted on several Pyrex specimens having factory-polished surfaces. These tests were valuable in refining the operation of the control assembly. They also provided information regarding contact resistance and parasitic heat flows, but nothing that was not better determined using the specimens described below. Hence, the results of these tests are not included in this report.

To evaluate contact resistance and, at the same time, study the effects of heat flow into the powder insulation, five specimens were prepared of thicknesses in the range 0.25 cm to 2.25 cm. All specimens were cut from the same piece of Pyrex and ground to a diameter of 2.540 ± 0.001 cm. The contacting surfaces were optically polished to be flat to within 1/10 fringe of sodium light (approx. 2.5×10^{-6} cm) and parallel to within 0.001 cm. Four of these specimens are shown in Figure 3.

3.2 Test Results for Pyrex Glass

Previous to the tests mentioned in Section 3.1, it was thought that heat exchanges between the bars and the powder insulation would be minimized if the guard temperatures were matched to the bar temperatures at points near the specimen. The mathematical analysis given in Appendix A showed, however, that matching these temperatures does not yield a minimum heat exchange with the insulation, because of longitudinal heat flow in the insulation. This analysis indicated the possibility of a superior procedure, i.e., to raise the hot guard to a temperature greater than that of the hot bar and to control the cold guard at a temperature less than that of the cold bar. The mathematical analysis predicts that at properly selected guard temperatures, the heat flow through the specimen will be equal to the measured power input. All tests discussed hereafter utilize this procedure. Because the exact guard temperatures desired cannot always be obtained in practice, corrections are made for the small amount of parasitic heat flow calculated mathematically (see Appendices A and B).

Representative results obtained in the study of contact thermal resistance are shown in Figure 4. The total resistance per unit area, R_T , is plotted against specimen thickness, L . The four curves shown represent specimen mean temperatures of 200°, 300°, 400°, and 500°C, respectively. For the tests shown, diatomaceous earth was used as insulation, the pressure exerted on the specimen was 10 kg/cm² (approx. 142 psi), and the specimen temperature gradient was approximately 25°C/cm.

The total thermal resistance per unit area should plot linearly versus the specimen thickness, provided the contact resistance is substantially the same at a given temperature and the conductivity of the specimens does not vary. Under these conditions, the points for a given mean temperature should fall on a straight line having an ordinate intercept equal to the total contact resistance of the two interfaces. The inverse slope of the line is equal to the thermal conductivity of the specimen at the mean temperature concerned.

The lines shown are least mean square lines fitted for the four specimens 0.75 cm and greater in length. The data obtained at 200° and 300°C conform closely to straight lines for all five specimens, while at 400° and 500°C there is more departure from linearity. The resistance of the 0.25-cm specimen is substantially greater than that indicated by the corresponding line, which may be due to one or both of two possible causes: a) the error involved in determining

a small temperature difference as the difference between two high measured temperatures, b) a decrease in radiative heat transfer near the ends of a specimen, which becomes proportionately more significant as specimens become shorter, and as the specimen mean temperature increases.

Because of the uncertainty of the resistances obtained for the 0.25 cm specimen, the lines were drawn ignoring them. However, at 200° and 300°C, the points for this specimen conform closely to the respective lines, although the departure becomes greater at 400° and 500°C.

The ordinate intercepts of the lines, which should indicate the total contact resistance, are in fair agreement for the 200°, 300°, and 400°C lines. The intercept for the 500°C line is markedly greater, which would not be expected, since the contact resistance theoretically should diminish slightly with increasing temperature. It is thought that this large intercept implies inexactness in the resistances obtained at 500°C, which with our present information is ascribed to the effect of significant radiative heat transfer in the specimens at 500°C.

The inverse slopes of the four lines give the apparent conductivity (including radiation) of the Pyrex material, which was determined from these measurements as

Temperature	200°	300°	400°	500°
App. Cond., mw/cm-°C	13.3	14.1	16.4	20.7

For the reason given above, the apparent conductivity at 500°C is questionable.

Many materials such as alumina become non-opaque to thermal radiation at elevated temperatures and therefore it was felt that more investigation of the radiation component of heat transfer at the lower temperatures possible with Pyrex would be helpful in analyzing the relative conductive and radiative components of heat transfer in other materials at elevated temperatures. For an infinite body, the radiation conductivity has been derived independently by several investigators and is given in the literature as

$$k_r = \frac{16}{3} \frac{n^2 \sigma T^3}{\alpha}$$

where n = the index of refraction of the medium
 σ = Stefan-Boltzmann constant
 T = absolute temperature
 α = absorption coefficient.

For short specimens such as those being investigated in this project, the problem becomes much more complex. In the specimen, near the hot and cold bars, the radiative heat transfer depends on the emittances of the contacting surfaces and throughout the specimen is a function of both the temperature and the temperature gradient in the specimen.

In order to investigate the effect of contact surface emittance on the apparent conductivity, a series of tests was conducted on a specimen of 1.257-cm thickness. The tests were made at several mean temperatures from 200° to 550°C with the specimen in contact with (a) the oxidized stainless steel faces of the hot and cold bars, (b) 1-mil shiny annealed platinum foil, and (c) 1-mil platinized (blackened) annealed platinum foil.

In this series of tests, the insulation used was diatomaceous earth, the pressure applied was 10 kg/cm², and the temperature gradient across the specimen was approximately 25°C/cm. The results are shown in Figure 5. The "over-all thermal conductivity" of the specimen (which includes the effect of contact resistance) is plotted against temperature for each of the three curves shown. Judging from the relative positions of the three curves at the lower temperatures, the contact resistances were least for the platinized foil and greatest for the oxidized stainless steel. Although two additional interfaces were added when foil was used, better thermal contact appears to have been made because of the plasticity of the foil. The curve for the platinized foil lies above both of the other curves. The explanation of this is that the foil makes excellent contact, and radiation into the specimen is increased because of the higher surface emittance of the platinized foil.

Before giving further consideration to the results in Figure 5, it is necessary to have more information concerning contact resistance when annealed platinum foil is used as an interface medium. Accordingly, additional tests were made on a 0.252-cm specimen at 200°, 300°, 400°, and 500°C with a shiny foil interface, the results of which are shown in Figure 6 along with previous results obtained with the 1.257-cm specimen. Comparing Figure 6 and Figure 4, it is seen that the contact resistances were made smaller when shiny annealed platinum foil was introduced as an interface between the specimen and the oxidized stainless steel bars. Again, a relative increase of the ordinate intercept at 500°C was found, as was first seen in Figure 4.

Resuming consideration of the data of Figure 5, Figure 7 presents apparent conductivities obtained by making corrections for the contact resistances, using the values from

Figure 4 for the oxidized stainless steel interfaces, and those from Figure 6 for the bright platinum foil interfaces. Because contact resistances were not determined separately for the platinized foil, the conductivity in Figure 7 for that condition was adjusted to agree with that for the other curves at the lower temperatures. As higher temperatures are approached, the three curves diverge from a straight line and from each other. Curve A rises most rapidly, indicating a relatively large radiation component due to the high surface emittance of the oxidized stainless steel. Curve B is the flattest of the three curves, indicating a lower radiation component due to the low surface emittance of the shiny foil. Curve C, for platinized foil interfaces, lies between the other two curves, indicating that its emittance was intermediate (which probably was so, since only a very thin layer of platinum black was present on these foils). These three curves represent apparent thermal conductivities, including both conductive and radiative components as governed by the emittances of the interfaces. It must be emphasized that curves A, B, and C are valid only for specimens of 1.257-cm thickness under the conditions of the tests.

If the conductive component is assumed to be linear, as it appears to be at lower temperatures, curve D would represent the conductive thermal conductivity. Values of thermal conductivity for Pyrex glass taken from published literature agree with the curves shown in Figure 7 to within ± 10 percent over the temperature range shown. Some literature values¹ are indicated in Figure 8, along with curve D of Figure 7. A linear extrapolation of this curve agrees to within ± 3 percent of values near room temperature obtained in this laboratory using the NBS guarded hot plate (ASTM C-177), as indicated in Figure 8.

As a further comparison, the data from the table on page 7 are plotted in Figure 8 as open circles. These points, which represent the apparent conductivity (including radiation) of the glass, were obtained by taking the inverse slopes of the lines in Figure 6. It is seen that the agreement with the curve from Figure 7 is good at lower

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1. F. Birch and H. Clark, The thermal conductivity of rocks and its dependence upon temperature and composition, Am. J. Sci. 238, 529 (1940).
R. W. B. Stephens, The temperature variation of the thermal conductivity of Pyrex glass, Phil. Mag. 14, 897 (1932).
C. F. Lucks, H. W. Deem, and W. D. Wood, Thermal properties of six glasses and two graphites, Am. Ceram. Soc. Bul. 39, 313 (1960).

temperatures. The apparent conductivity at 500°C shows a substantial component of radiation conductivity.

Later developments and modifications to be discussed below indicate that the above data may have been subject to a systematic error, which probably increased with temperature. Thus, there may be inaccuracy in the absolute magnitudes obtained, but the qualitative discussion given above is considered valid.

4. MODIFIED PROTOTYPE DESIGN

As was mentioned above, when the prototype absolute cut-bar apparatus was first designed and built, it was believed that optimum guarding could be achieved by matching the temperature profile along the guard to that along the bars and the specimen. Later developments have shown, both theoretically and experimentally, that this procedure does not yield optimum guarding. As discussed in Section 3.2 and Appendix A, a more satisfactory procedure is to raise the hot guard to a temperature greater than that of the hot bar and to control the cold guard at a temperature less than that of the cold bar.

The design of the prototype apparatus rendered the attainment of these better guard temperatures difficult, especially at lower temperatures, because of the presence of metallic "bridges" between the hot and cold bars and their respective guards (see Figure 2).

To cope with this difficulty, the prototype was modified (see Figure 9). Metallic "bridges" were eliminated. Thin-walled stainless steel tubing was used to support the hot guard from below and the cold guard from above, and as a support for the hot bar. A ceramic spacer ring was used to separate the hot and cold guards. To enable free access to the specimen, the assembly, consisting of the guards, spacer ring, surrounding powder insulation, and case, was mounted so that it could be readily lowered away from the hot and cold bars.

5. RESULTS OBTAINED USING THE MODIFIED APPARATUS

In order to check the modified prototype apparatus in respect to heat flow and temperature measurement, a series of comparison tests was conducted. Two materials were selected: (a) a metal whose conductivity had been determined by an independent absolute longitudinal heat flow method; (b) the 1.257-cm sample of Pyrex No. 7740, which had been measured in the apparatus before it was modified.

A specimen of nickel-chrome alloy (see Figure 10) was fabricated from the same slab of material used to fabricate a specimen measured in the NBS metals apparatus. The specimen was installed in the absolute cut-bar apparatus without an interface material. A stainless steel spacing ring was installed between the hot and cold guards and diatomaceous earth was used as insulation.

The thermocouples shown on the centerline of the specimen were installed in ceramic tubing inserted in drill holes to the center of the specimen. The thermocouples shown at the edge of the specimen were peened into small holes in the surface of the specimen.

Conductivity determinations were made at eight temperatures ranging from about 115° to 800°C. The thermal conductivity was calculated separately for each of the four thermocouple spans indicated in Figure 10. The results of these calculations are plotted in Figure 11. The circles refer to the peened thermocouples. The solid line represents a quadratic equation of least squares fit through all of the points obtained. It will be noted that the data for the peened thermocouples are much smoother than those for the inserted thermocouples. The dashed lines above and below the solid line bound the region plus and minus 5 percent of the solid line.

In Figure 12, the data of Figure 11 have been replotted as departures from the smoothed data obtained using the NBS metals apparatus. The superiority of the peened thermocouples is quite evident in this plot. It is seen that the agreement of the absolute cut-bar apparatus with the NBS metals apparatus is excellent.

These data for the nickel-chrome alloy indicate that the absolute cut-bar apparatus is capable of accurate thermal conductivity measurements if the temperature gradient in the specimen can be precisely determined. The absolute power measurement appears to be satisfactory and the parasitic heat flows, both through the insulation and through the hot bar support, are adequately evaluated.

It should be noted, however, that this nickel-chrome specimen is not similar to the specimens for which this apparatus is designed. In the measurements previously described for Pyrex glass prior to modification of the apparatus, thermocouples were not placed in the specimens. The temperature gradient in these specimens was evaluated by means of thermocouples located in the hot and cold bars. In order to determine this gradient, it was necessary to evaluate thermal contact resistance by means of an elaborate

series of measurements on specimens of several thicknesses. Although the data acquired for Pyrex appear to have internal consistency, there remains the possibility of a systematic error in determining the temperature gradient. This will be further discussed below.

The thermal conductivity of thermoelectric materials must be as low as possible if the thermoelectric device is to have a high efficiency. As was the case for Pyrex, the heat flow through a specimen of low conductivity will be much less than that through the nickel-chrome specimen. Consequently, the evaluation of parasitic heat exchanges assumes far greater importance for these low conductivity specimens.

The purpose of the above discussion is to point out that the measurement of thermal conductivity is much more difficult for thermal semiconductors than for metals. The excellent agreement of the data for the nickel-chrome alloy with that from the NBS metals apparatus should not be taken as proof of this apparatus and method for materials having a conductivity much closer to that of the surrounding insulation. It is our belief that the method will prove to be more than adequate for such materials, but it must be emphasized that the agreement of the nickel-chrome data with another absolute method and the internal consistency of the Pyrex results are not sufficient proof of the method. Further tests are necessary to establish the accuracy of the method for low conductivity materials.

In order to check the modified prototype apparatus on materials of low conductivity, the 1.257-cm specimen of Pyrex glass No. 7740, which had been measured in the apparatus before it was modified, was installed. Diatomaceous earth was used as insulation, the pressure on the specimen was 10 kg/cm^2 , and the temperature gradient was about 25°C/cm . No interfacial material was used. Conductivity determinations were made at temperatures ranging from 100° to 500°C . An attempt was made to duplicate as closely as possible the test conditions attained for this specimen prior to modification of the prototype apparatus.

The results obtained on this specimen of Pyrex did not agree with those obtained previously and discussed under Section 3.2. More scatter was observed in the data than before. An extrapolation of the newly acquired data to room temperature agreed substantially with the previous room temperature extrapolation and with the values obtained using the NBS guarded not plate (see Figure 8). However, the slope of the thermal conductivity curve was more nearly that of the NBS guarded hot plate, or that of Lucks, Deem, and Wood. This resulted in a thermal conductivity value that

was 10 or 15 percent lower at 300°C than the values previously obtained (tentative data of Figure 8).

Numerous checks and tests were taken in an effort to isolate this difficulty. No instrumentation difficulties were found. The data for the nickel-chrome alloy indicated that the power measurement was satisfactory. It was therefore concluded that the temperature gradient in the specimen was in question.

Since the thermal conductivity of the stainless steel hot and cold bars was known closely, it was possible to compute the temperature gradients in the bars by using this conductivity and the measured power input in the formula

$$\frac{\Delta T}{\Delta z}_{ss} = \frac{Q}{K_{ss}A}$$

where $\Delta T/\Delta z$ is the temperature gradient in the hot or cold bar

Q is the measured power input corrected for extraneous heat exchanges

K_{ss} is the thermal conductivity of the stainless steel bars

A is the cross-sectional area of the bars.

The results of this calculation were not in satisfactory agreement with the bar temperature gradients as determined by the inserted thermocouples in the bars. This would seem to indicate that the temperatures in the bars were not known accurately, and hence the temperature gradient through the Pyrex specimen was in error. A similar analysis of the earlier data discussed under Section 3.2 indicated that there may have been errors in determining the specimen temperature gradient prior to modification of the prototype apparatus.

Owing to the difficulties of temperature gradient measurement, it was not possible to determine which, if any, of the data acquired for Pyrex glass are correct. As was stated at the end of Section 3.2, the qualitative conclusions drawn in that section are considered to be correct. However, the thermal conductivities shown in Figure 7 and Figure 8 may be in quantitative error.

6. ADDITIONAL RESULTS

6.1 Addition of Thermocouples

A further change in the apparatus was made in view of the results obtained above. As was noted, the data acquired from the peened thermocouples (Fig. 11 and 12) are much

smoother than those for the inserted thermocouples. Consequently, it was decided to install peened thermocouples in the hot and cold bars of the prototype apparatus in an effort to better determine temperatures in these bars.

The thermocouples shown on the centerline of the bars in Figure 9 are the original thermocouples which were installed in two-hole ceramic tubing inserted to the center of the bars. The thermocouples shown at the surface of the hot and cold bars are the new chromel-alumel thermocouples which were peened into small holes in the surface of the bars.

6.2 Measurements on Pyroceram

Owing to the difficulties associated with thermal radiation through the Pyrex glass specimen, it was decided to undertake thermal conductivity determinations on small specimens of an opaque material. Two specimens of Pyroceram Code 9606 were prepared having thicknesses of approximately 1.26 cm and 0.77 cm, respectively. These specimens were not optically polished, but had a fine surface grind on the contacting faces.

Determinations were made on the two specimens at mean temperatures of approximately 200°, 300°, 400°, 500°, and 600°C. For these tests, diatomaceous earth was used as insulation, the pressure on the specimen was 10 kg/cm², and the temperature gradient was 20° to 25°C/cm. Platinized foil was used as an interfacial material to improve contact. The results of these tests are shown in Figure 13. The lower two curves show apparent thermal conductivity plotted against temperature for the two Pyroceram specimens. The limits shown above and below each datum point indicate the variation of the conductivity if various combinations of the thermocouples in the hot and cold bars are used to determine the temperature drop across the specimen; the data points are the arithmetic averages of the three values obtained in each test.

The upper curve in Figure 13 displays the thermal conductivity of Pyroceram Code 9606 after contact resistance adjustments are made to the lower curves. In making these contact resistance adjustments, it was assumed that the contact resistances were the same for the two specimens. The platinized foils were not in very good condition, especially for the tests on the 0.77-cm specimen, so that the assumption may not be strictly correct. For a material of low conductivity, such as Pyrex, the contact resistance adjustment is small, so that an error would not seriously affect the resulting conductivity value. However, for a material

of higher conductivity, like Pyroceram, an error in contact resistance evaluation would more seriously affect the conductivity values. The conductivity shown for Pyroceram is therefore considered tentative.

A series of measurements was conducted on a 0.77-cm specimen of a commercial alumina. The data acquired were smooth from 200° to 600°C. Dense alumina has a high thermal conductivity in this temperature range. Consequently, contact resistance adjustments are very large. Comparing the results of these measurements with literature values for similar material, it was observed that the resistance of the two contacts exceeded that of the specimen. Accurate determination of such relatively large contact resistances would require accurate thermal resistance measurements on specimens of several thicknesses. It is questionable whether or not contact resistance would reproduce sufficiently well from one specimen to the next to enable precise evaluation. Due to the uncertainty of contact resistance adjustment, the data for the 0.77-cm alumina specimen are not presented in this report.

An investigation of the temperature gradients in the hot and cold bars was made for both the inserted and the peened thermocouples. The peened thermocouples were noted to be superior, but were not entirely satisfactory. At least part of this difficulty is attributed to the use of chromel-alumel thermocouple wire.

7. FUTURE ACTIVITIES

It is felt that sufficient information has been acquired in the period covered by this report to permit design and construction of the high temperature model of the absolute cut-bar apparatus. Design work on this apparatus has been substantially completed and construction has begun.

The design of the high temperature model will parallel that of the modified prototype apparatus. The hot and cold bars will be fabricated from 60% platinum-40% rhodium alloy. The remaining high temperature structural portions of the apparatus are to be constructed of a high purity (+99%) alumina. All heaters will be 80% platinum-20% rhodium alloy. Thermocouples are to be reference grade platinum/platinum-10% rhodium.

In order to check the apparatus when it is completed, it has been decided to conduct measurements on two materials of known thermal conductivity. The nickel-chrome alloy measured in the modified prototype apparatus will be the high conductivity material. For the low conductivity

specimen, a material having a thermal conductivity similar to that of many thermoelectric materials would be preferable. The measurements made on Pyroceram seem to indicate that it would be a satisfactory material. A specimen of Pyroceram Code 9606 is currently being fabricated for measurement in the NBS metals apparatus. Specimens from the same bar will be measured in the high temperature absolute cut-bar apparatus. Dr. D. C. Ginnings of the Heat Measurements Section, NBS, intends to measure the thermal diffusivity of this material sometime soon, thus providing additional valuable cross-checks.

It has been pointed out in this report that the two quantities which must be determined in a steady-state measurement of thermal conductivity are the heat flux through the specimen and the temperature gradient in the specimen. For the absolute cut-bar method, the results shown and the analysis of heat flow given in Appendix A indicate that the heat flux through the specimen can be determined with sufficient accuracy. It is felt, however, that the temperature gradient in the specimen has not been adequately determined for the measurements given in this report. It was originally felt that measurements could be made expeditiously, utilizing thermocouples exterior to the specimen and making corrections for contact resistance. This has not proved to be the case. The contact resistance is too large, and possibly too uncertain, to be handled in this manner, even with the use of optically flat surfaces.

In the apparatus presently being constructed, a different method of measuring specimen temperature gradients will be attempted. Thermocouples will be affixed in small grooves cut in the convex surface of the specimen, and these thermocouples will be utilized to determine the temperature gradient. In order to assure uniform heat flow, contact resistance will be reduced by spraying the ends of the specimen with platinum. It is hoped that these modifications will enable more accurate determination of temperature gradients, and thus remove the remaining largest source of inaccuracy involved in these measurements.

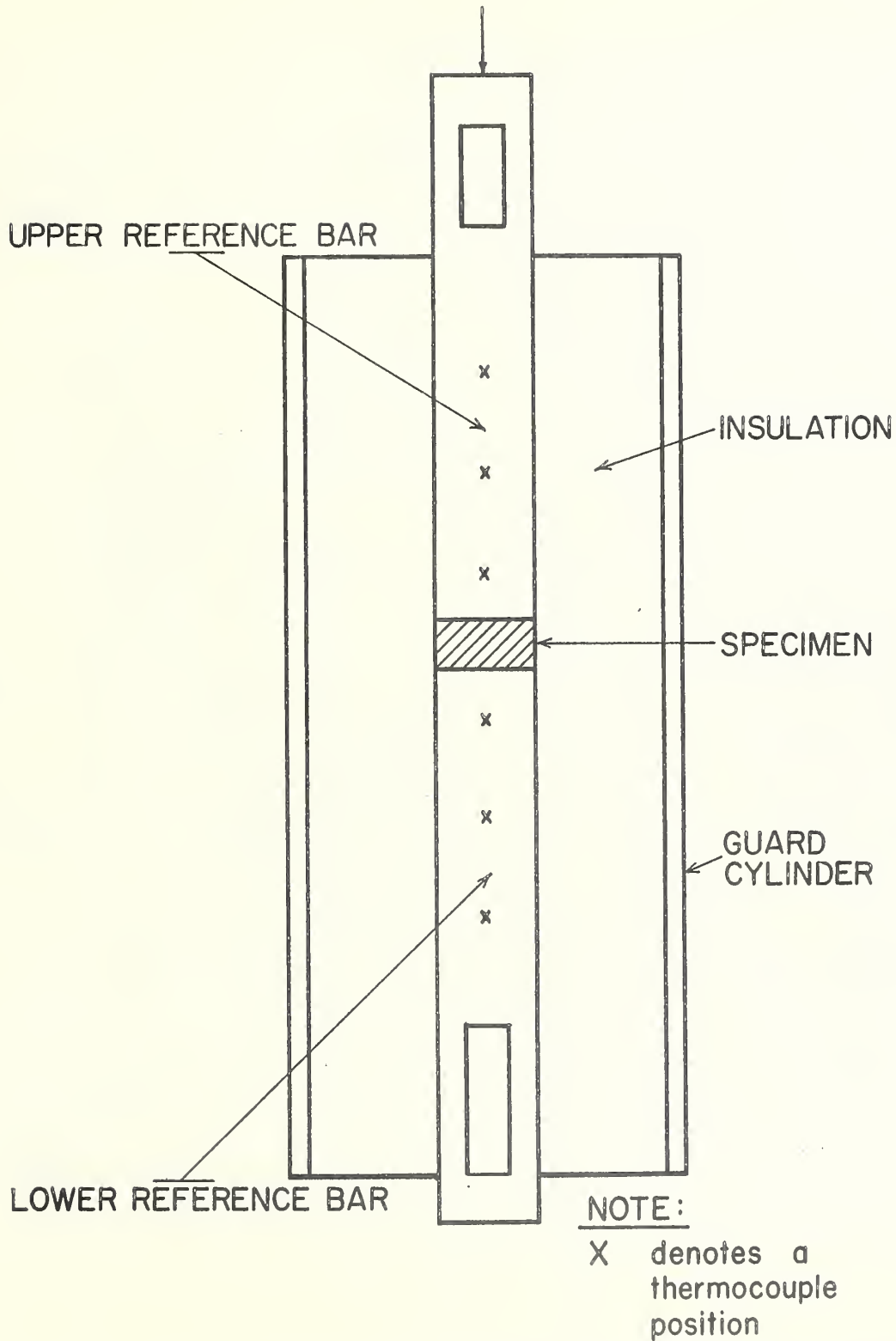


Fig. 1. A schematic cut-bar apparatus utilizing reference bars to determine heat flow through the specimens.

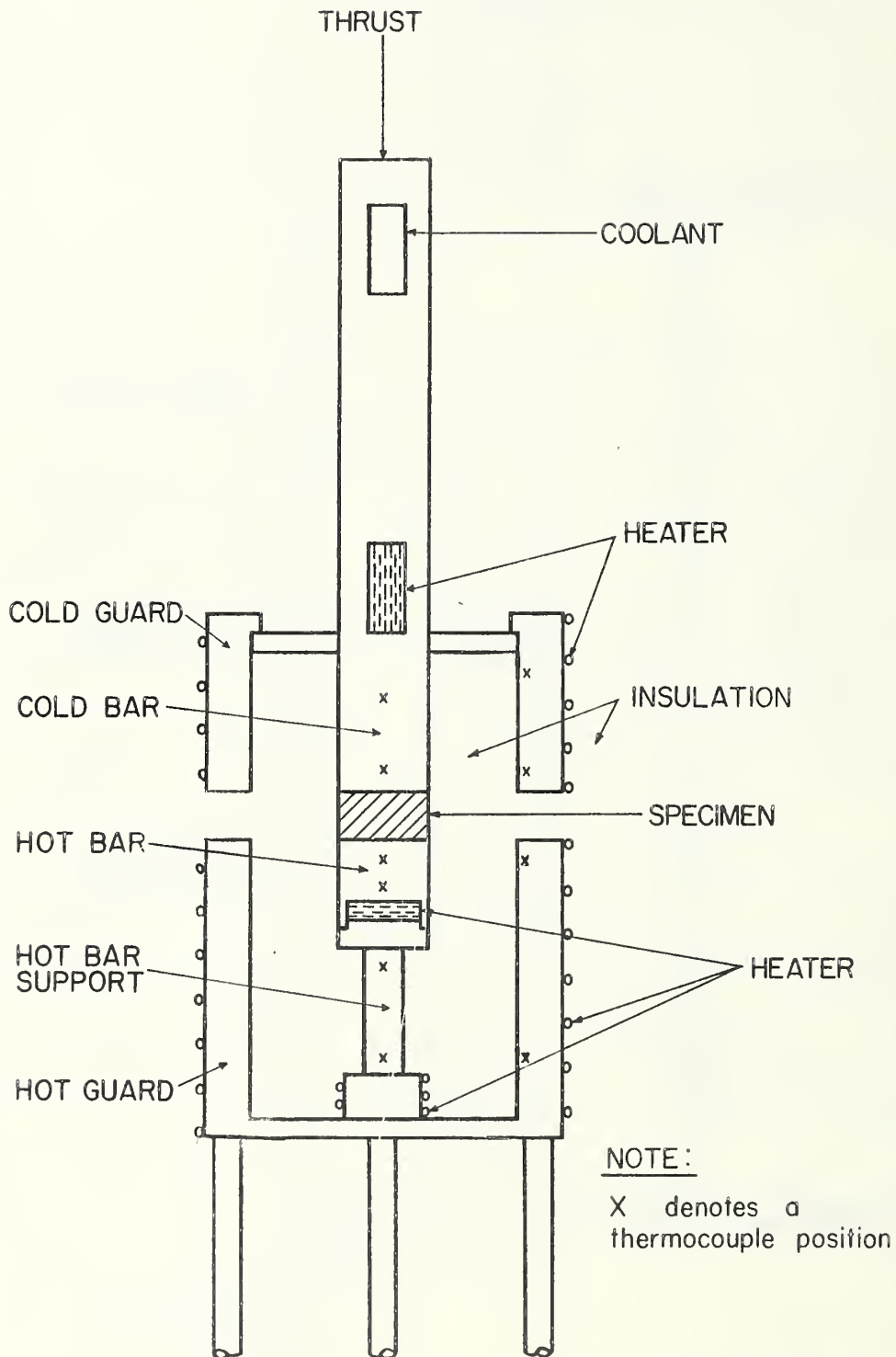


Fig. 2. The original prototype absolute cut-bar apparatus utilizing the measured power input to an electrical heater to determine heat flow through the specimen.

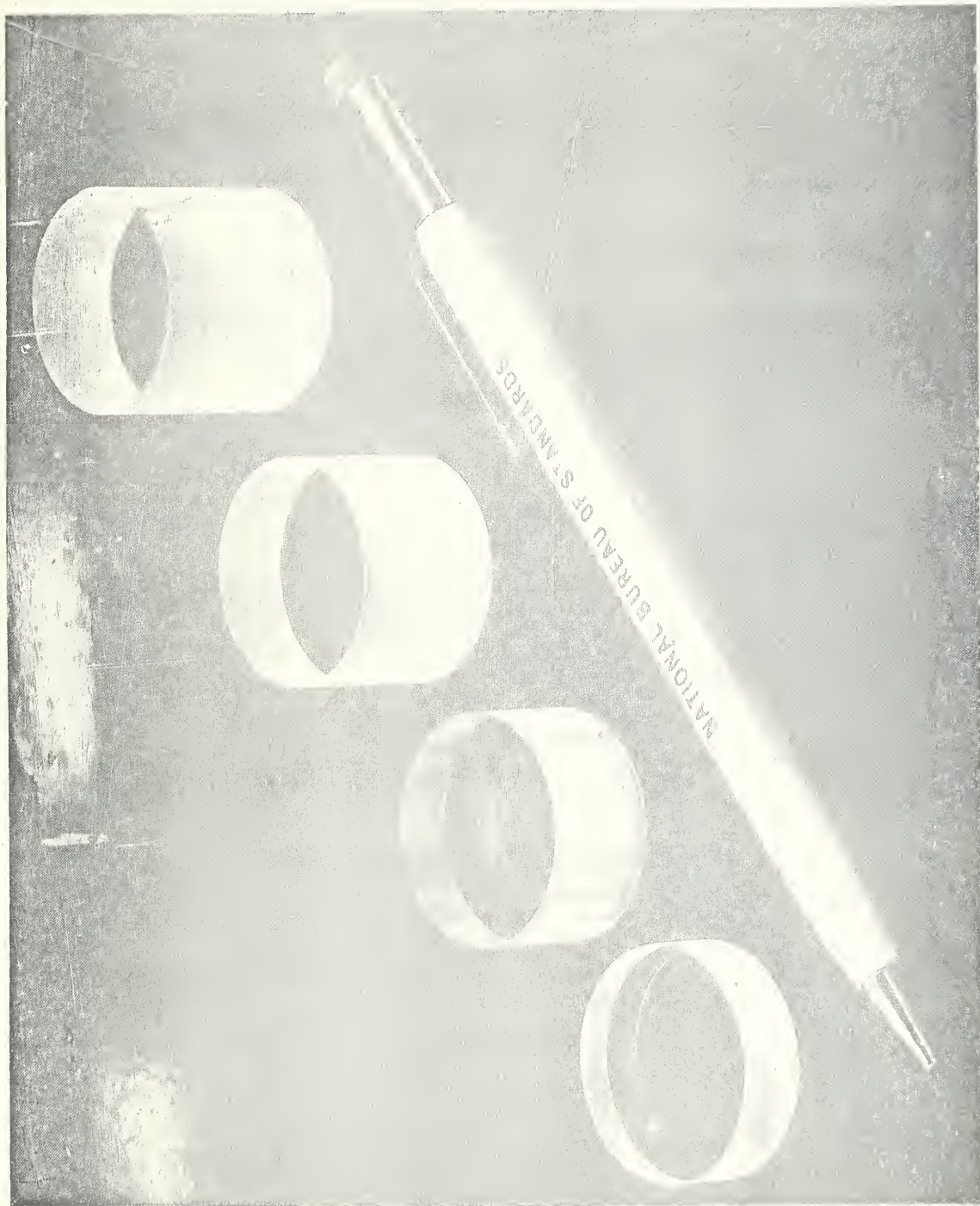


Fig. 3. Four of the Pyrex glass No. 7740 specimens used in the evaluation of contact resistance.

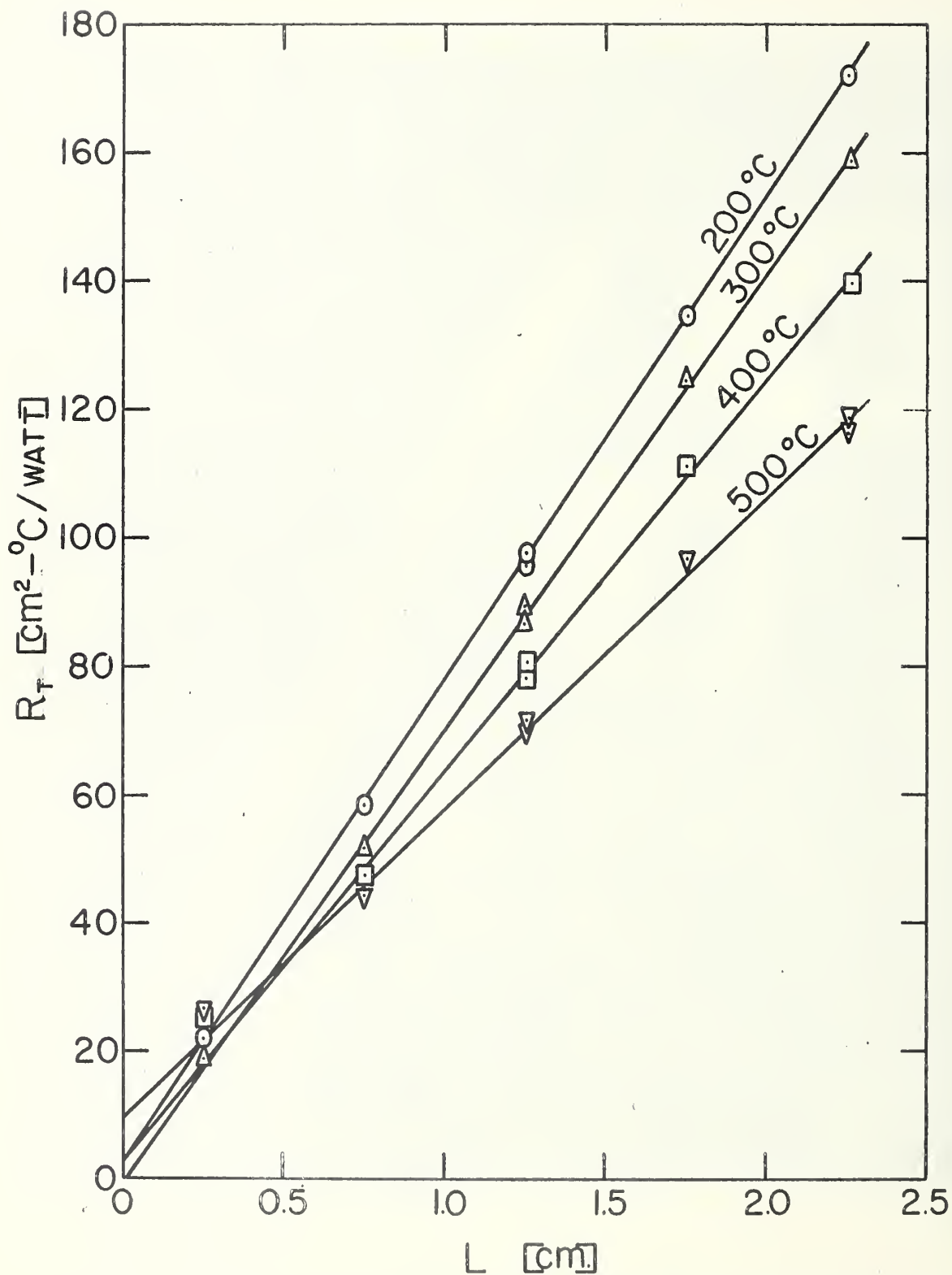


Fig. 4. Total thermal resistance, including contact resistance, for several specimens of Pyrex glass No. 7740.

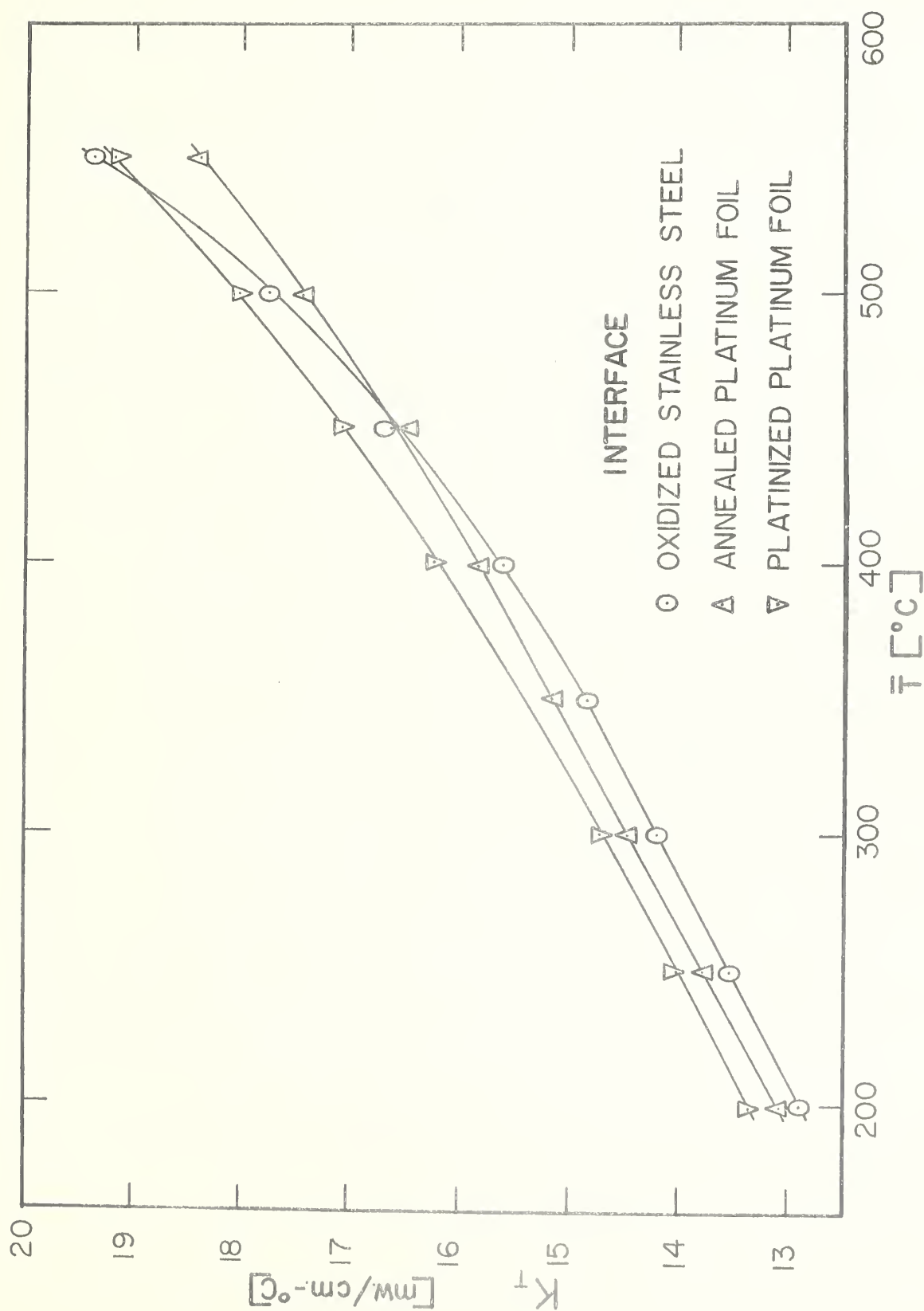


Fig. 5. Over-all thermal conductivity of Pyrex glass No. 7740, no corrections having been made for the contact resistance of three types of interfaces.

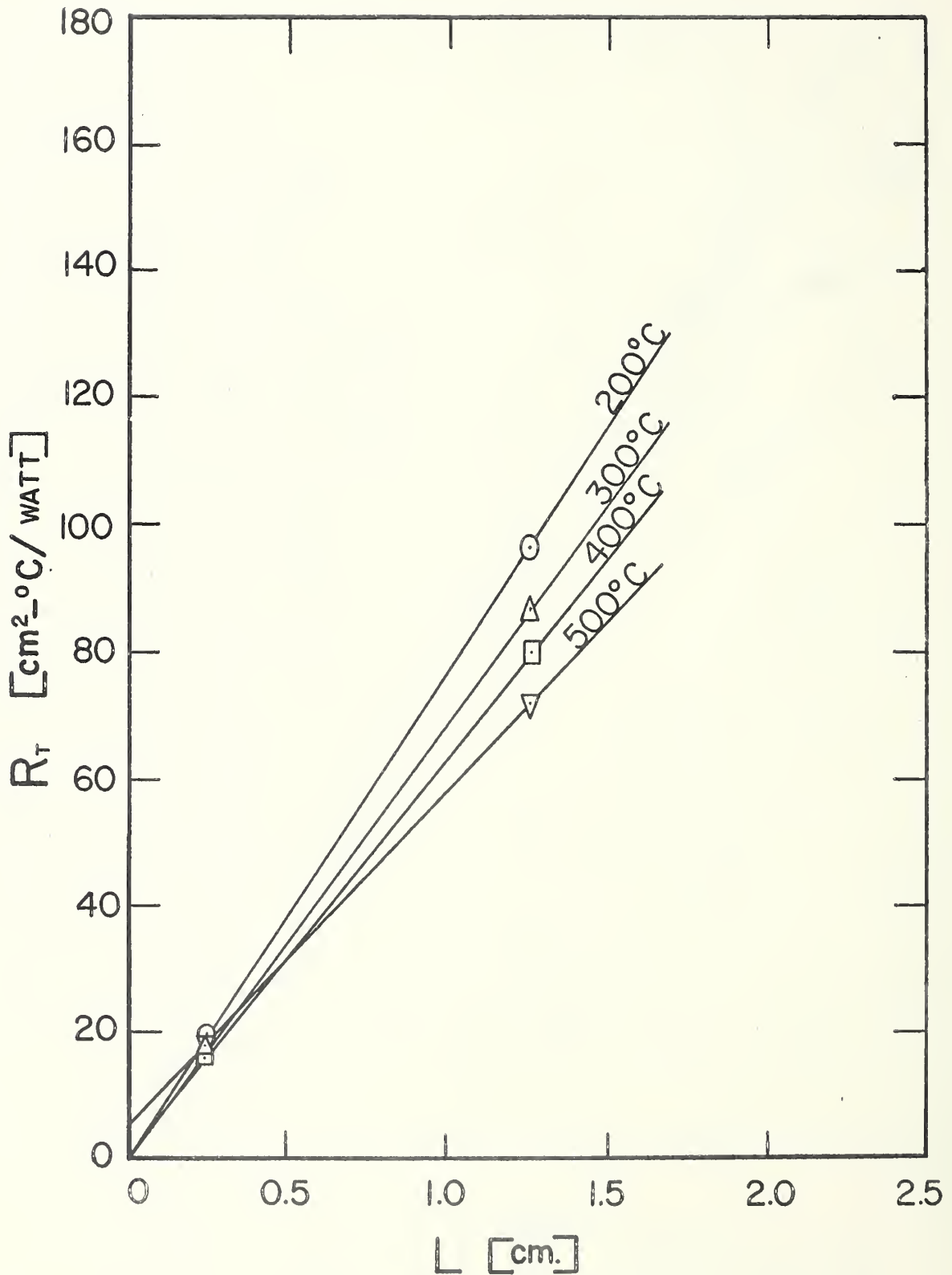


Fig. 6. Total thermal resistance, including contact resistance of platinum foil interfaces, of two specimens of Pyrex glass No. 7740.

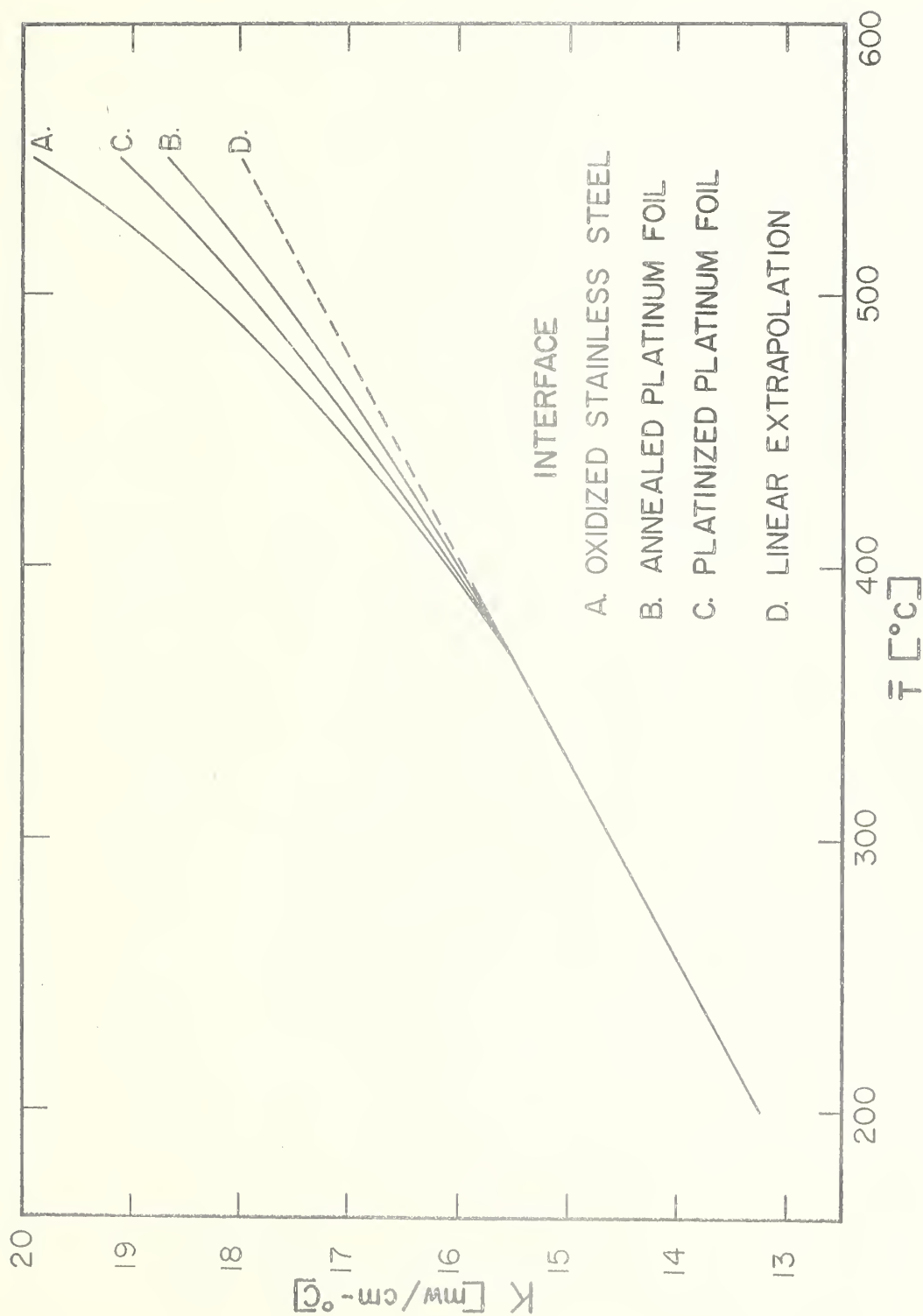


Fig. 7. Thermal conductivity of Pyrex glass No. 7740, as a function of temperature, including corrections for contact resistance.

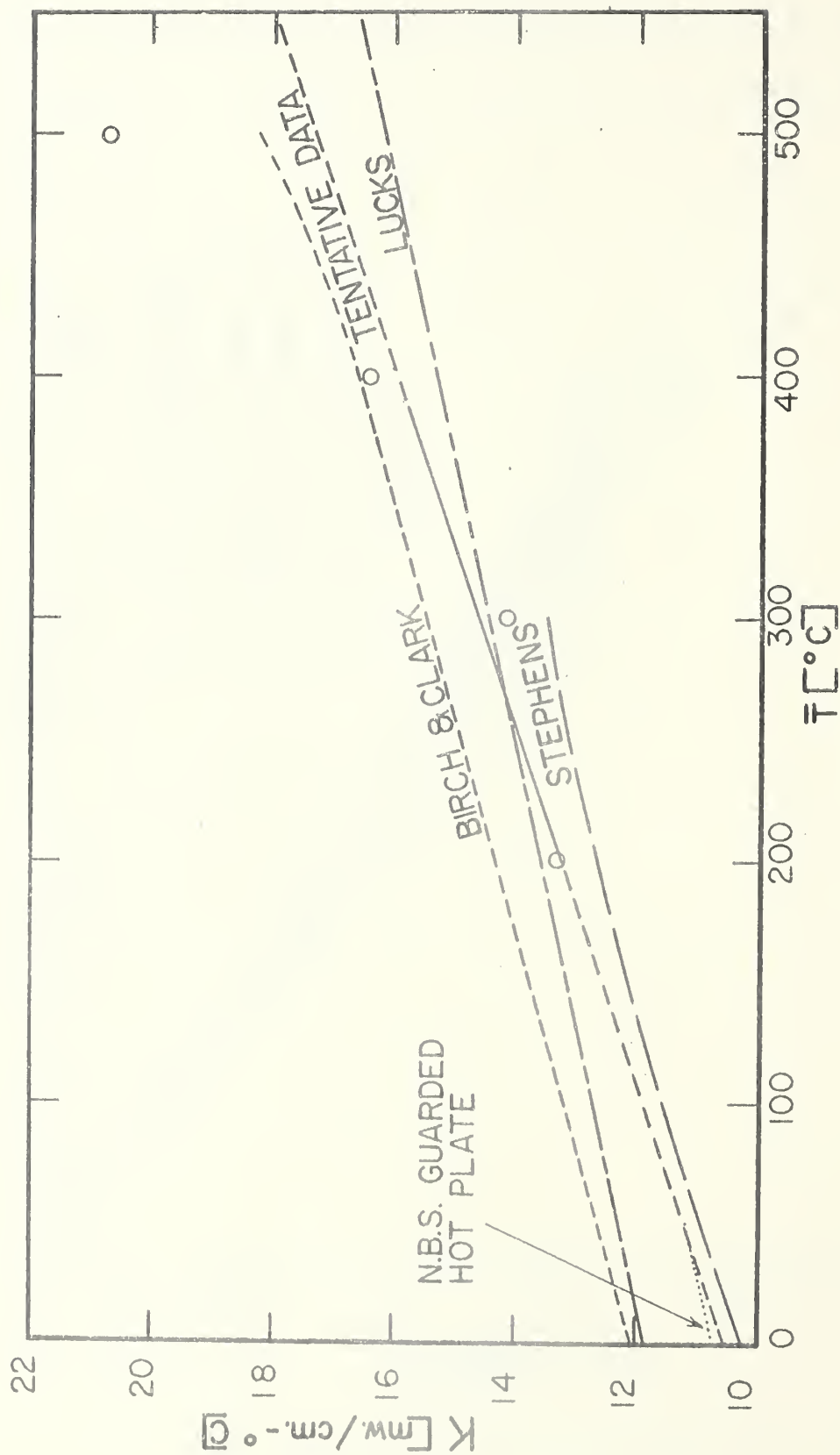


Fig. 8. Thermal conductivity of Pyrex glass No. 7740 (tentative data) compared with literature values. The circles indicate additional results derived from the data of Figure 4.

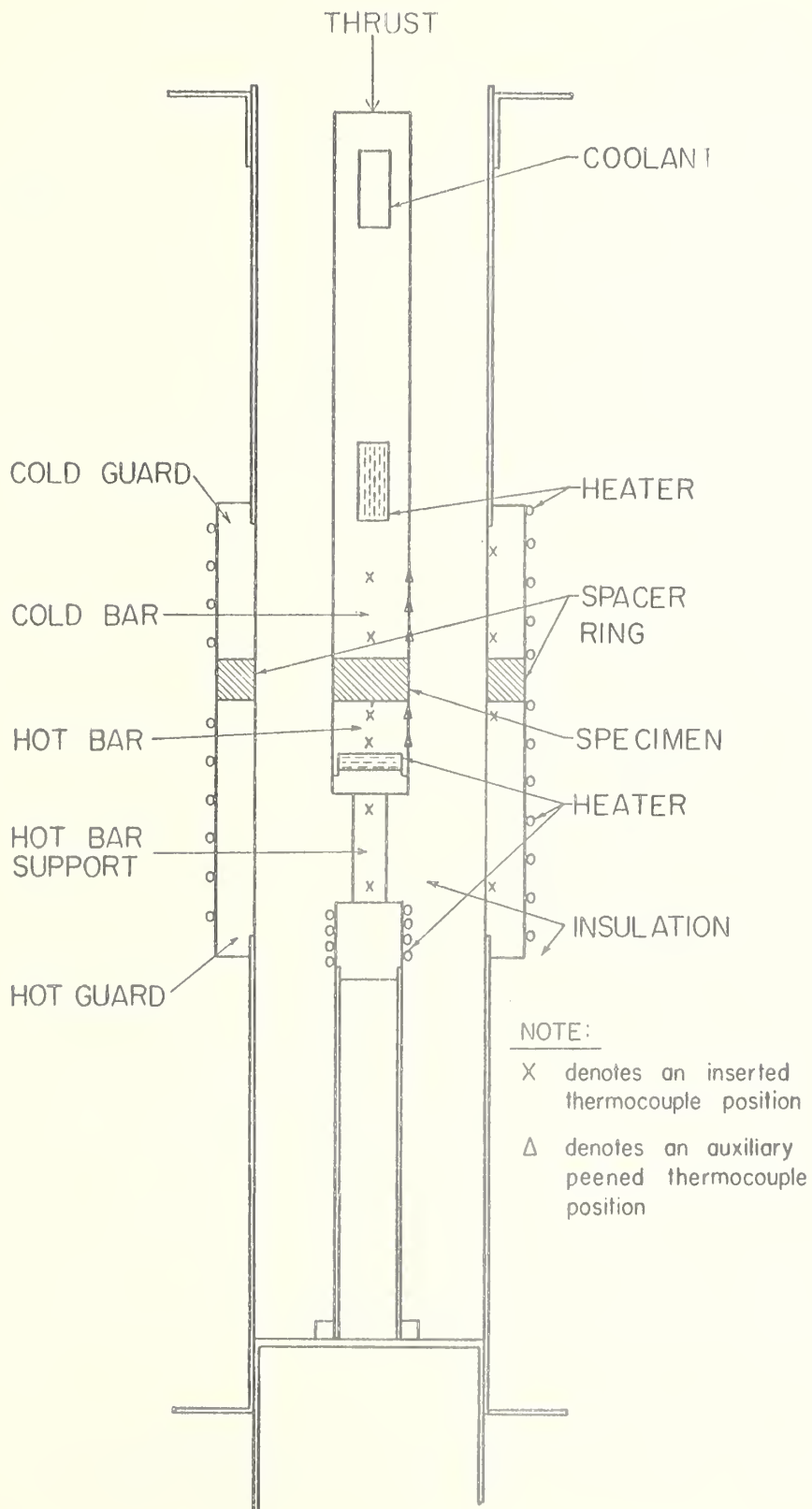
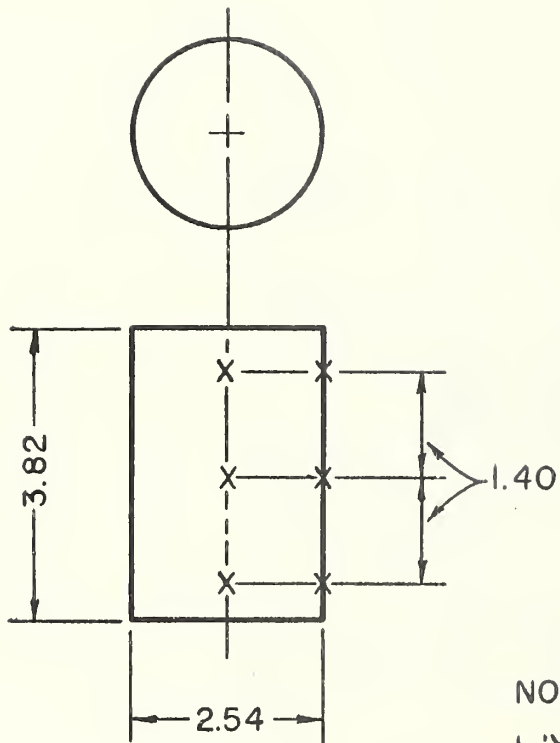


Fig. 9. The modified prototype absolute cut-bar apparatus.



NOTES:

1. 'X' DENOTES THERMOCOUPLE POSITION.

2. LENGTHS GIVEN IN CM.

Fig. 10. The nickel-chrome alloy specimen used to check the modified prototype apparatus.

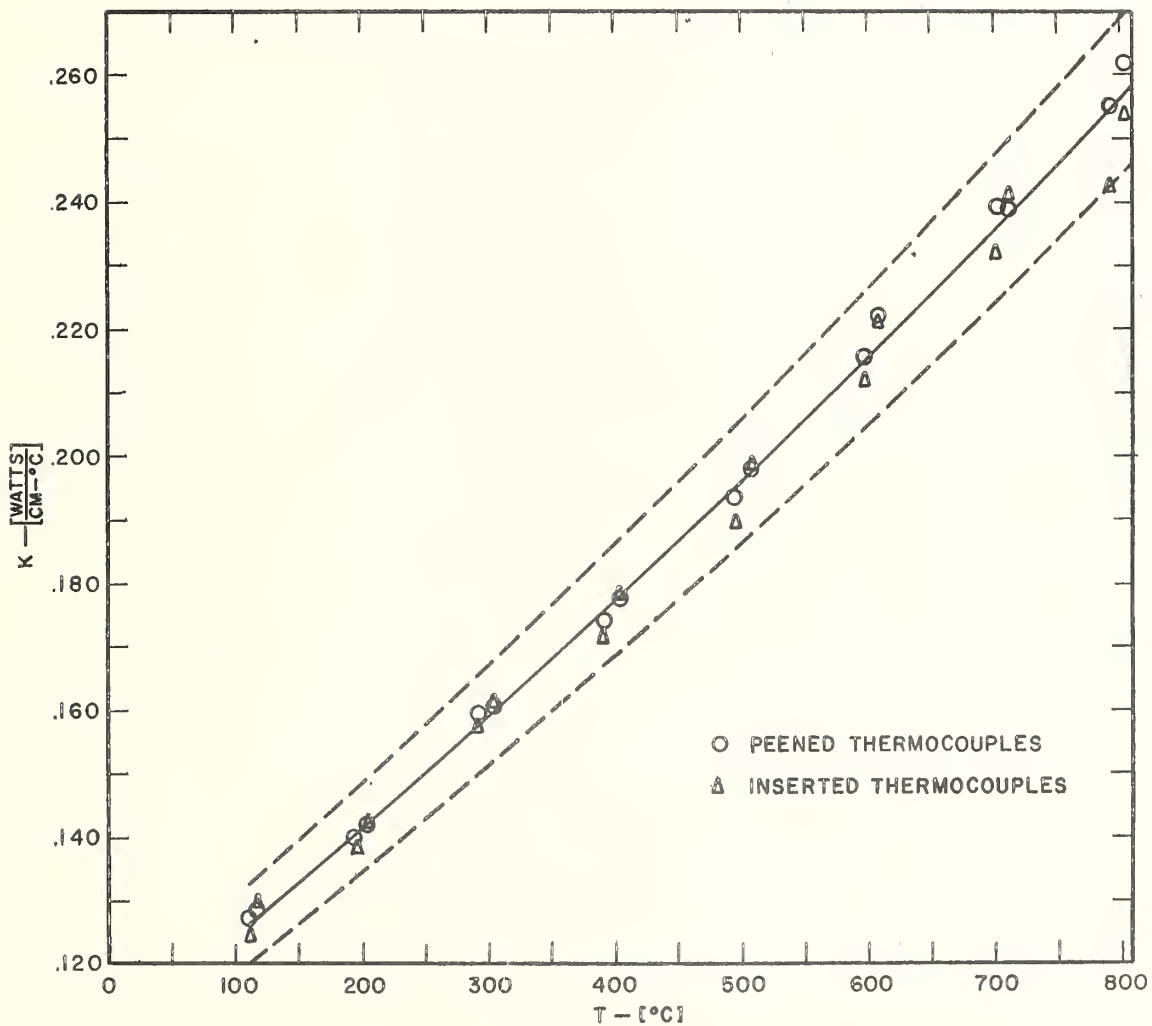


Fig. 11. Thermal conductivity of a nickel-chrome alloy as determined using two methods of thermocouple installation in the specimen.

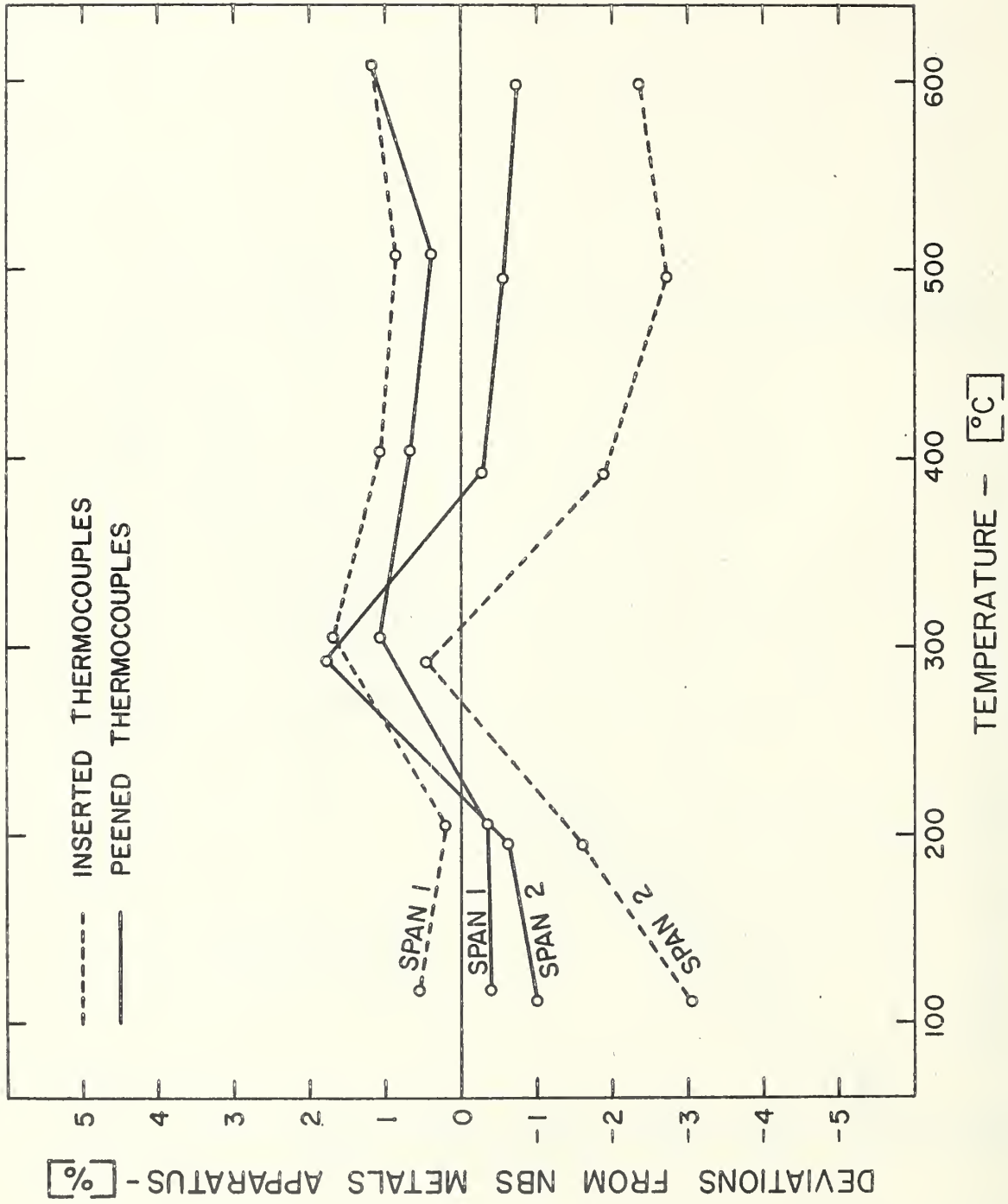


Fig. 12. Departures of the data for the nickel-chrome alloy from the smoothed data from the NBS metals apparatus.

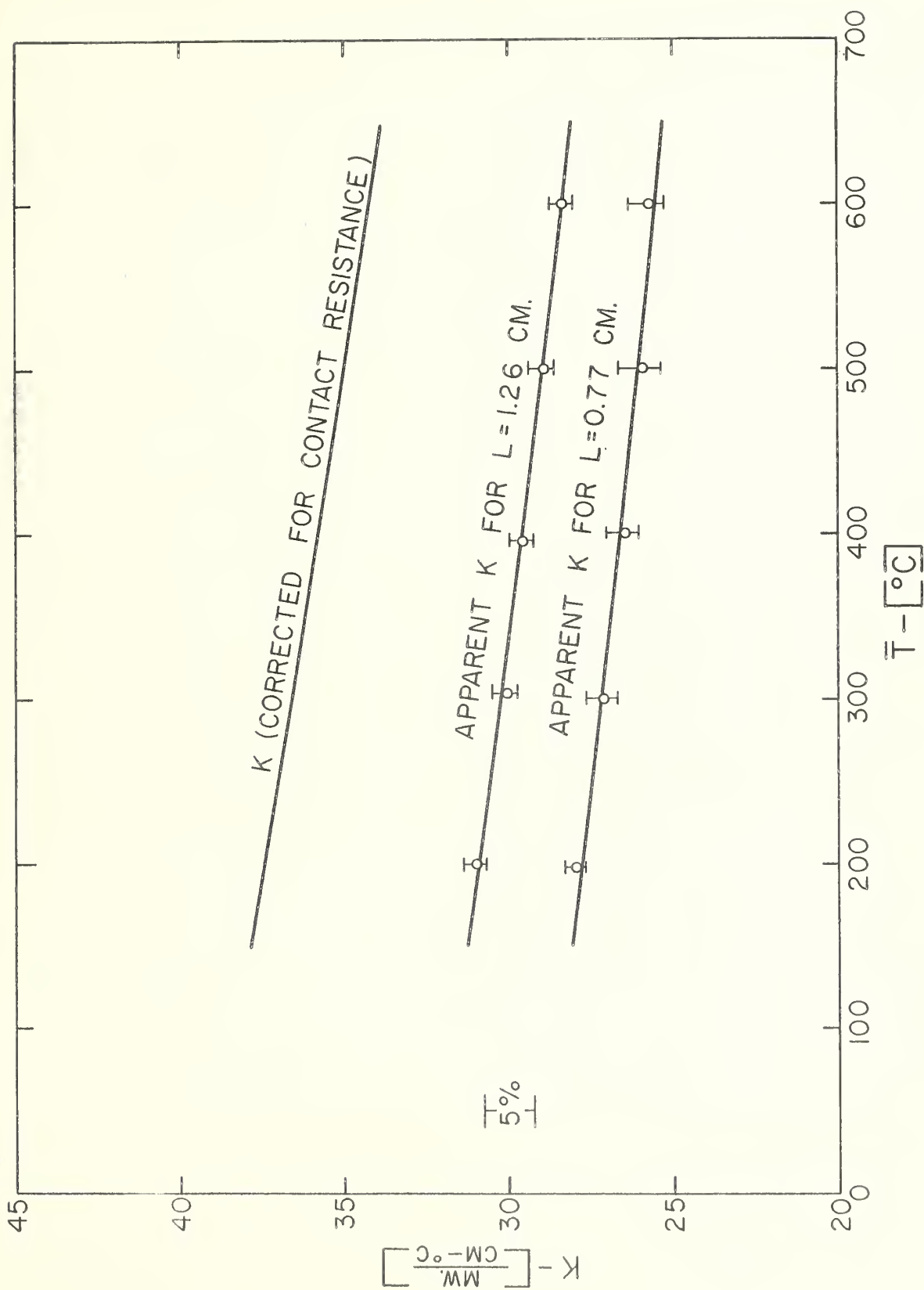


Fig. 13. Thermal conductivity of Pyrocera Code 9606 versus temperature, before and after correcting for contact resistance.

APPENDIX A

GUARDING OF A THERMAL CONDUCTIVITY APPARATUS
OF THE CUT-BAR TYPE

The general physical arrangement of a comparative "cut-bar" apparatus for determination of thermal conductivity was shown in Figure 1. The modified prototype absolute "cut-bar" apparatus, shown in Figure 9, has a similar physical arrangement. It is the purpose of this discussion to consider heat exchanges between the bars and specimen and the surrounding powder insulation in an apparatus of this type.

Some insight into this problem can be achieved by consideration of a rather crude model. The hot bar, specimen, and cold bar can be thought of as three thermal resistances in series. Shunting each of these resistances is a resistance consisting of a portion of the surrounding powder insulation. For the case in which the thermal conductivity of the specimen is different from that of the bars, there will be a corresponding change in the longitudinal temperature gradient in the adjacent insulation. Since the thermal conductivity of the insulation does not change materially, the insulation must exchange heat with the bars and specimen. In particular, if the specimen has a lower conductivity than the bars, it is necessary for the insulation to steal heat from the bars and specimen on the hotter side and return this heat on the cooler side.

It is also necessary to originally establish a longitudinal gradient in the insulation near the hotter bar. In the design shown in Figure 1, this gradient will be established near the lower end of the bar so that there will be no further exchange of heat until the specimen is approached. However, in the case where the bars are quite short the longitudinal gradient may not become established, so that heat exchanges occur along the entire length of the system. Even in the case where the specimen and the bars have the same conductivity, there may be heat interchanges along the entire system. In order to study the parasitic heat flows mentioned, it is necessary to perform a rather extensive analysis.

Although there may be both radial and longitudinal heat flows in the powder insulation, it is the radial gradient at the surface of the bars and specimen that is of particular interest. In the ideal case, the radial gradient at this surface would be zero. This is, however, not the case in general, so that an analysis of the heat flow across

this surface must be made. The heat flow across this surface in a longitudinal element of area will be

$$dq = 2\pi a k \left(\frac{\partial \theta}{\partial r} \right)_{r=a} dz \quad (A1)$$

where a is the radius of the bars and specimen
 k is the thermal conductivity of the powder insulation
 θ is the temperature in the system
 r is the radial coordinate
 z is the longitudinal coordinate.

The net power flowing across the surface $r=a$ between $z=z_1$ and $z=z_2$ is

$$P_{12} = 2\pi a \int_{z_1}^{z_2} k \left(\frac{\partial \theta}{\partial r} \right)_{r=a} dz \quad (A2)$$

In order to evaluate this integral it is necessary to have a knowledge of the thermal conductivity of the powder insulation and the temperature distribution within the insulation. An exact treatment of the system requires solution of the four body composite system involving the two bars, the specimen, and the powder insulation. Mathematically, this problem is intractable because of the many continuity conditions which must be satisfied.

For this apparatus the region consisting of the powder insulation was analyzed as a boundary-value problem. The measured surface temperatures along the bars and the guard cylinders were used to establish simple linear functions representing the temperatures on the curved surfaces of the region. The ends of the region were assumed to be closed by logarithmic radial temperature distributions, which provide temperature continuity. The analytical solution of the temperature distribution in the region established by these boundary conditions was utilized to evaluate $(\partial \theta / \partial r)_{r=a}$ in the integral in Eq. (A2). The thermal conductivity of the powder insulation was measured in another apparatus.

Since the analytical solution of this problem is in the form of an infinite Fourier series, it was necessary to employ the use of a digital computer for numerical evaluation of the data. The details of this mathematical development are not amenable to brief treatment and hence have been excluded from this report. It is planned to publish the mathematical analysis of a cut-bar thermal conductivity apparatus soon.

Prior to the mathematical analysis outlined above, it was thought that heat exchanges between the bars and the powder insulation would be minimized if the same temperature distribution was maintained along the guard cylinder as along the reference bars and specimen, so that guard temperatures matched those of the bars at corresponding longitudinal positions. It was soon found that such a procedure does not eliminate extraneous heat exchanges between the bars and specimen and the surrounding powder insulation. For a Pyrex glass specimen 1/2 inch thick at 500°C, using diatomaceous earth as insulation in the prototype apparatus, heat flow through the specimen was about 13 percent less than the measured power input for a test in which guard temperatures approximately matched those in the bars. It is seen that the matched-guard condition does not reduce extraneous heat flows to the small magnitude desirable for good accuracy of measurement.

For a material such as glass, or many semiconductor materials having a thermal conductivity much less than that of the bars, there will be a radial heat flow from the hotter bar near the specimen to establish the larger longitudinal gradient necessary in the adjacent insulation. This radial flow can be greatly reduced by raising the temperature of the guard immediately opposite. Similarly, the heat flow from the insulation into the colder bar can be lessened by lowering the temperature of the guard opposite the colder bar. What this does, in effect, is to permit the guard cylinder to set up most of the longitudinal gradient in the powder insulation adjacent to the bars and specimen. The exact temperature distribution that should be placed on the guard cylinder has not been determined. Theoretically, it should be possible to guard the system so that the radial gradient at the surface of the bars and specimen is exactly zero. Such a guarding condition would be almost impossible to achieve experimentally at elevated temperatures.

What was done in practice was to establish a compromise temperature distribution along the guards to yield a small radial heat flow from the bars and specimen. By astute adjustment of the guards, it is possible to make the heat flow through the specimen closely equal to the measured power input at the heater. Although there are small heat gains and losses along the assembly, the net total of these interchanges can be made nearly zero, for the length of the assembly up to the specimen, by means of suitable guard temperatures.

For example, for the 1/2-inch Pyrex glass specimen at 500°C, using diatomaceous earth as insulation in the prototype apparatus, heat flow through the specimen was within

1/2 of 1 percent of the measured power input using the compromise guarding condition described above. Furthermore, the mathematical analysis corrects for even this small error.

All of the data presented in this report were computed using the above mentioned analysis to evaluate parasitic heat flows. It is believed that, by the use of such an analysis, the precision and accuracy of the heat flow determinations were greatly improved.

APPENDIX B

CALCULATION PROCEDURE

The numerical calculation of the data was effected by means of a digital computer. The over-all thermal conductivity of the specimen, including the contacts, was calculated using the following equation.

$$k = \frac{\bar{Q}L}{A\Delta T} \quad (B1)$$

where k = thermal conductivity (watts $\text{cm}^{-1} \text{ deg}^{-1} \text{ C}$) at temperature T

\bar{T} = mean temperature of specimen ($^{\circ}\text{C}$)

L = specimen thickness (cm) at temperature \bar{T}

A = specimen cross-sectional area (cm^2) at temperature \bar{T}

ΔT = temperature difference (deg C) across specimen and contacts

\bar{Q} = average heat-flow (watts) through the specimen.

This equation is valid for steady-state longitudinal heat flow over a small temperature interval. The determination of the various factors in the conductivity equation will now be discussed individually.

1. Specimen thickness (L).

The specimen thicknesses were carefully measured at room temperature, using a metric micrometer which could be read to 0.0005 cm. For the nickel-chrome alloy, the distance between thermocouple junctions was used. Thickness as used in Eq. (1) was determined by the following:

$$L = L_{25}[1 + \alpha(\bar{T} - 25)] \quad (B2)$$

where L_{25} is the measured thickness (cm) at room temperature (25°C)

α is the coefficient of linear thermal expansion ($\text{deg}^{-1} \text{ C}$) of the specimen material

2. Specimen cross-sectional area (A).

The diameter of each specimen was carefully measured at room temperature and the area computed. Cross-sectional area as used in Eq. (1) was determined by the following

$$A = A_{25}[1 + \alpha(T - 25)]^2 \quad (B3)$$

where A_{25} is the area (cm^2) at room temperature.

3. Temperature difference $[\Delta T]$.

The temperature difference across the specimen is determined by measuring two temperatures in the hot bar and extrapolating linearly to determine the temperature of the face of the hot bar, and similarly measuring two temperatures in the cold bar and extrapolating linearly to determine the temperature of the face of the cold bar. The difference between these temperatures represents the temperature drop across the specimen and the two interfaces. The temperature difference as used in Eq. (B1) was determined by the following:

$$\Delta T = T_H - T_C \quad (\text{B4})$$

where T_H is the inferred temperature ($^{\circ}\text{C}$) of the hot bar face
 T_C is the inferred temperature ($^{\circ}\text{C}$) of the cold bar face.

T_H and T_C were determined by linear extrapolation of temperatures measured in the hot and cold bars, respectively. For the measurements on Pyroceram and alumina, various combinations of the bar thermocouples were used.

4. Heat flow (Q).

The power input to the hot-bar heater was measured by means of a precision volt box and shunt box in conjunction with a d-c precision potentiometer. Corrections were made for the small current flow through the volt box.

The heat produced could flow, if permitted, in several directions:

- a) Up the hot bar, through the specimen and into the cold bar
- b) Up or down the hot bar support column
- c) Across the convex surfaces of the hot bar and the specimen.

These three classifications can best be treated separately:

- a) Ideally, the heat should flow entirely through the specimen. The heat flows b) and c) should therefore be minimized.

b) The heat flow up or down the support column was minimized by keeping the chromel-alumel differential thermocouple located in the column as nearly as possible at a zero reading. For tests in which the reading of the differential thermocouple was not zero, a correction was made for the slight heat flow through the support column. The area of metal and its conductivity, and the area of porcelain tubing and its conductivity, gave a value for the thermal conductance between the differential junctions. The reading of the differential thermocouple was used to compute the heat flow up or down the support column. This correction was confirmed experimentally.

c) The heat flow across the convex surfaces of the hot bar and the specimen was analyzed as presented in Appendix A. This analysis was utilized to determine the net heat flow across the inner surface of the hollow cylinder of powder insulation, taking place up to various longitudinal positions of interest on the assembly.

The net heat gain or loss across the convex surface was evaluated at both ends of the specimen and at the center of the specimen. In short, Eq. (A2) was used to compute the net heat interchange with the powder insulation over the region of the bar and specimen assembly up to selected longitudinal positions z_2 . These were chosen to correspond to the positions of the two ends of the specimen, and its center, and the weighted average of these three heat interchanges yields the average net gain or loss of heat, \bar{p} , between the heater and the specimen.

The heat flow as used in Eq. (1) was determined by the following

$$\bar{Q} = P + C\Delta t + \bar{p} \quad (B5)$$

where P is the measured power input (watts)

k is the conductivity of the powder insulation
(watts-cm⁻¹-deg⁻¹C)

a is the radius of the specimen (cm)

\bar{p} is the weighted average of the net heat interchange from the assembly for the specimen

Δt is the temperature difference in the support column as indicated by the differential thermocouple (deg C)

C is the heat flow up or down the support column due to a unit temperature difference (watts-deg⁻¹C).

U. S. DEPARTMENT OF COMMERCE

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